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DEVELOPMENT OF A FIRE TEST FACILITY FOR GRAPHITE FIBER-REINFORCED COMPOSITES

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PREFACE

This report summarizes work performed at Avco Specialty Materials Division, Lowell, Massachusetts, from August 1978 through September 1979 under Contract NAS1-15511 for NASA Langley Research Center. The project reported herein resulted in the modification and upgrading of Avco's Model 25 Fire Facility to provide capability for evaluation of the fiber release characteristics of composite materials in fire environments.

This program was conducted under the cognizance of Mr. Robert Jewell and Dr. Vernon Bell of the NASA LRC. The principal investigator was Mr. Glen Alexander. Mr. Ed Janas greatly influenced the design of many features of the facility and developed several test techniques which were essential to its successful operation.

The use of commercial products or names of manufacturers in this report does not constitute official endorsement of such products or manufacturers, either expressed or implied, by the National Aeronautics and Space Administration.

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I. INTRODUCTION AND SUMMARY

Recent interest in the development of graphite fiber-reinforced composites with improved resistance to aircraft crash fire environments leads to a requirement for defining suitable test techniques for quantitative evaluation of fiber release and for timely and economic screening of potentially improved composites. The test method requires simulation of the thermal parameters and combustion chemistry of aircraft crash fires and sufficient structural disturbance of the sample to cause dissemination of fiber material. Recovery of substantially all solid residue is desired, particularly that component consisting of small single fibers which would be readily distributed by air currents in an actual fire.

The Avco Model 25 fire simulation facility, located at Lowell, Massachusetts, is applicable to this type of activity. It has been in operation for nearly ten years in support of Avco's development of fire protection materials. The facility has been utilized for more than two thousand tests to evaluate a variety of materials in a closely controlled, well characterized fire environment which reproduces the thermal parameters and chemistry of a wide range of fires.

Under the current program, a number of modifications were added to the basic facility to make it specifically applicable for composite material screening tests. Most significant was the development of hardware for trapping fibers released during test and isolating them for quantitative measurement. Capability was added for increasing test section velocities and increasing the range of air/fuel ratios available from very rich to

very lean. A provision was added for agitation of the test specimen and the combustion gases by a pulsating gas supply. A summary of the salient features of the Model 25 facility as currently configured is presented in Table 1.

A variety of specimen configurations was evaluated by an extensive series of tests on a graphite-epoxy "reference composite" which is representative of material currently used in advanced aircraft. A standard test technique and specimen configuration were established which was satisfactory for the reference composite. A test program was then performed on several "alternate composite" materials to verify the validity of the test method for relative evaluation of a variety of composite materials. A fiber oxidation study was also performed to assess the significance of combustion of the fiber component in accounting for the quantity of residual material recovered after the test.

TABLE I

FIRE FACILITY FEATURES

FORCED GAS DRAFT THROUGH TEST SECTION AT VELOCITY TO 7.62 M/S (25 FT/SEC).

RADIANT SOURCE TEMPERATURE TO 1477^{o}K (2200^{o}F).

NATURAL GAS BURNER SUPPLYING METERED GAS RATES TO 8.5 M^3/HR (300 SCFH).

METERED AIR SUPPLY TO 85 M^3/HR (3000 SCFH).

AIR/FUEL MIXTURE RATIO READILY VARIABLE FROM ABOUT 6 TO 20.

PROVISION FOR AGITATION BY PULSATING GAS SUPPLY.

FIBER TRAPPING BY WATER SCRUBBER.

FIBER RECOVERY BY WATER FILTRATION.

CHROMEL-ALUMEL THERMOCOUPLE RECORDING AND MANUAL OPTICAL PYROMETRY AVAILABLE.

SPECIMEN PHOTOGRAPHY DURING FIRE EXPOSURE.

II. FACILITY DEVELOPMENT

The basic Avco Model 25 fire facility as it was originally configured is illustrated in Figure 1. It consisted of an electrically heated ceramic hood to provide a radiation heat source, a natural gas supply system to provide convective heating and combustion gases, and an opening in the floor for insertion of a test specimen mounted flush to the floor of the test section. In this configuration, the facility was utilized for more than two thousand tests to evaluate a variety of materials in a closely-controlled, well characterized fire environment which reproduces the actual temperatures and heat fluxes of a wide range of fires (References 1, 2, and 3).

Under sponsorship of the NASA Langley Research Center the facility was extensively modified to provide the capabilities necessary for evaluation of the fiber release characteristics of graphite-reinforced composites. The principal modifications included a forced draft exhaust system, a fiber trap, gas flow instrumentation, and a specimen agitation device. Specimen holders were made which permitted specimen orientation at angles to the flow as well as flush to the floor.

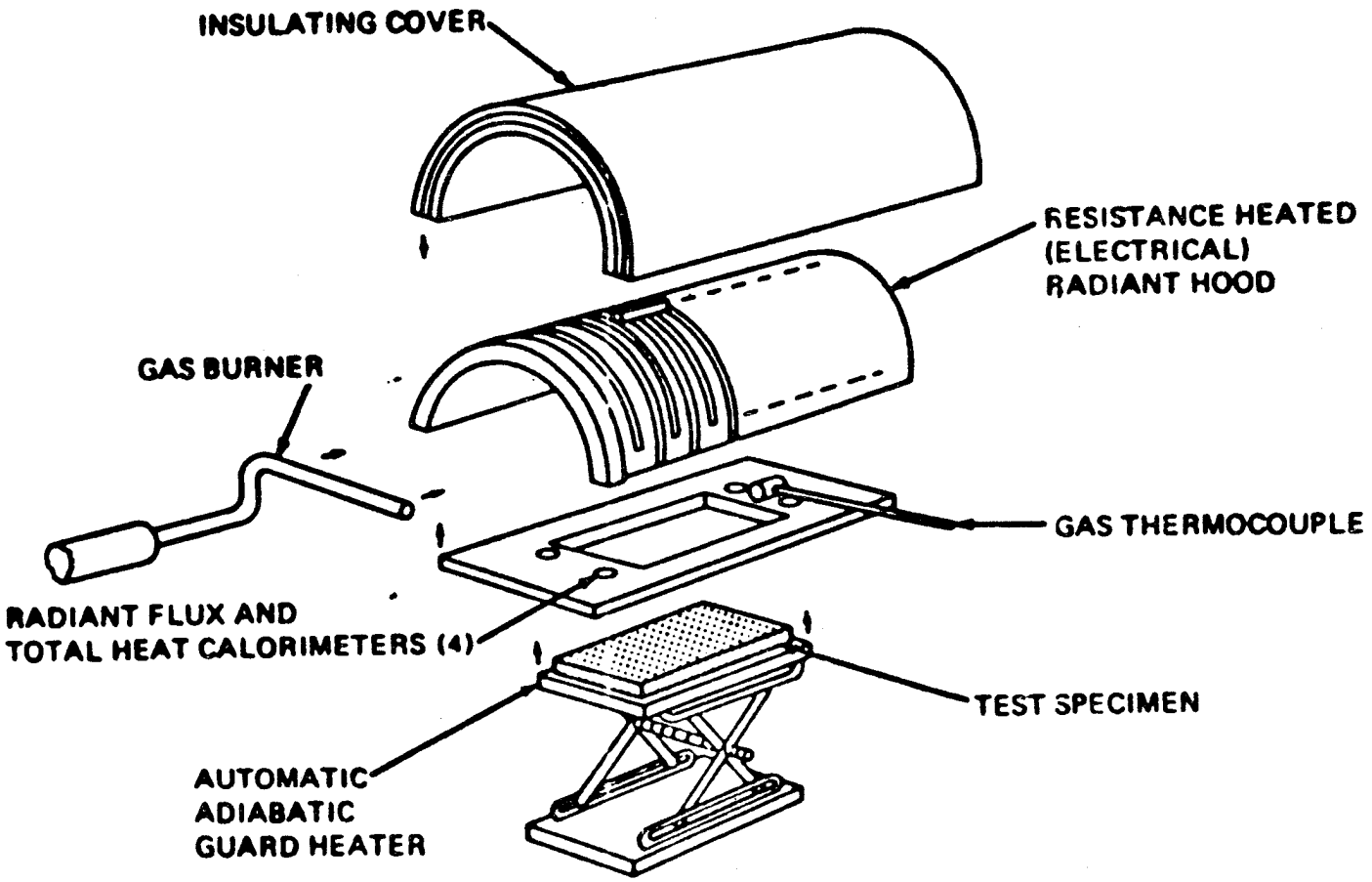


Figure 1. Original Model 25 Fire Facility Configuration

The final configuration evolved is illustrated schematically in Figure 2 and in the photograph of Figure 3. The exhaust is provided by a 1/2 H.P. centrifugal blower and is sufficient to draw 85 m^3/hr. air through the entire system.

The fiber trap is essentially a small water-bath air-scrubber made of stainless steel and operating at a water flow rate of 20 liters per minute. The combustion gases are passed through a water spray which is effective for cooling the gases to about 93°C. The gases are expanded into a plenum chamber and passed through a vertical water curtain which removes the fibers from the combustion gas. The fibers are then collected on a cellulosic filter through which the scrubber water is passed.

The water curtain effectively removes not only the graphite fibers but quantities of soot from composite resin products and from combustion of rich mixtures of fuel. For those tests where a soot-fiber mixture was collected on the filter, it was found that the soot could be made to pass through the filter by adding a moderate quantity of detergent to the mixture. Fiber samples collected in this manner from graphite-epoxy burns were quite clean. The fiber collected in the water trap included single fibers, lint or small clumps of fiber, and perhaps even a few very small fragments. Weight of fibers was deduced by weighing the fiber-loaded filter after drying and comparing with the pre-test weight of the filter. Fiber samples from typical tests range from 1/20 to 1/4 gram and were measured to the fourth decimal place with a Mettler micro-balance.

Experiments were performed to verify the effectiveness of the fiber trap by operating the system cold with a dry cellulosic filter installed at the exhaust blower inlet. Chopped graphite fibers, 2 mm long,

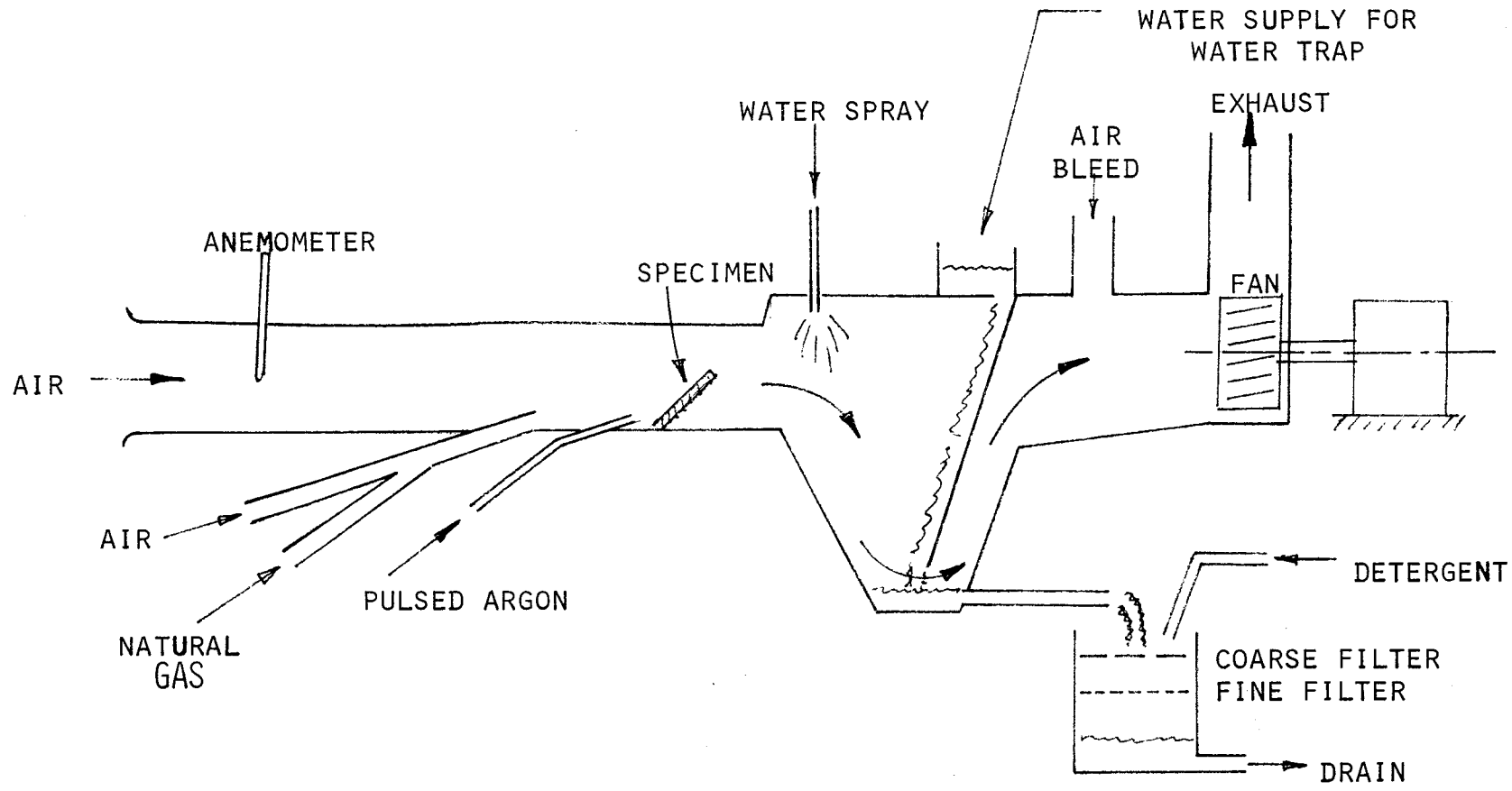


Figure 2. Facility Schematic

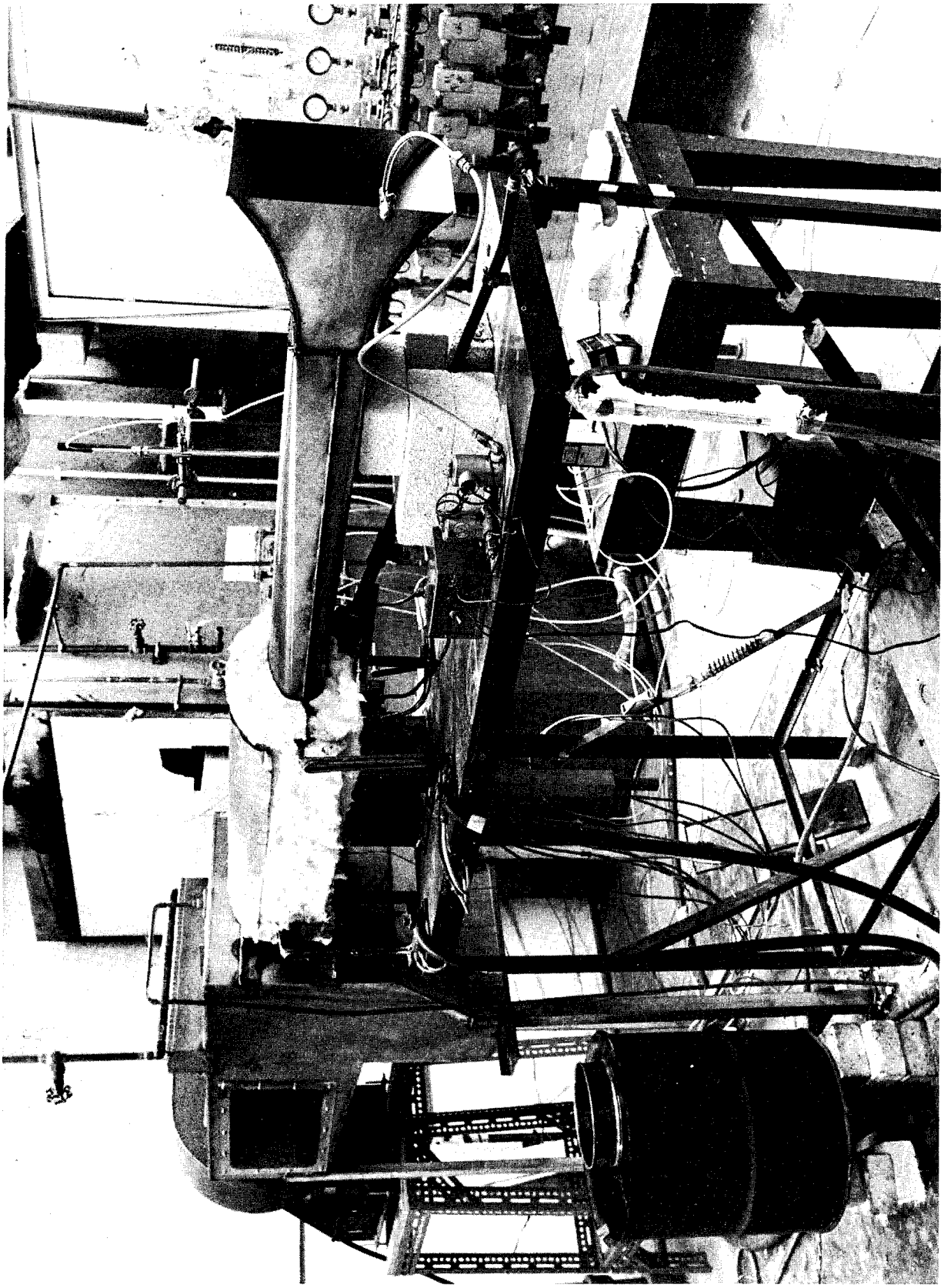


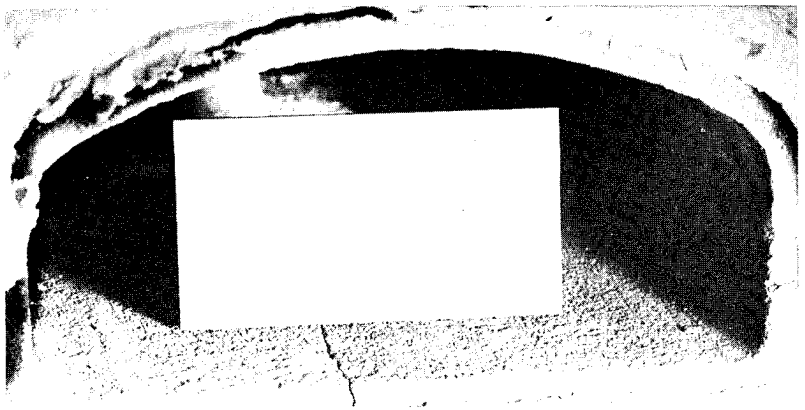
Figure 3. Modified Model 25 Facility

were introduced into the fiber trap with a paint-sprayer using a premix of fibers and water. No fibers were observed reaching the dry filter while substantial quantities were collected on the wet filter.

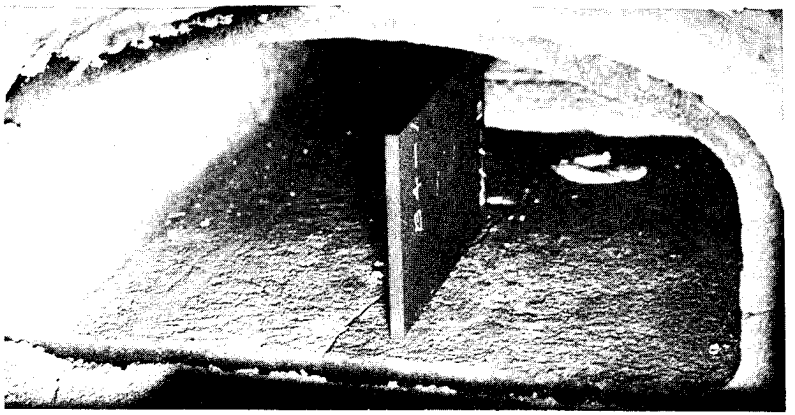
A number of flat-panel specimen configurations and orientations were evaluated in the course of developing a standardized test procedure (Figure 4). These included flush-mounted in the floor of the test section, vertically mounted to the floor and oriented parallel to the flow direction, vertically mounted and oriented perpendicular to flow, and oriented at 45 degrees to both the floor and the flow direction.

For flush mounting to the floor, the specimen dimensions are 124 x 124 mm. Any thickness can be accommodated. This configuration was found to be unsatisfactory for materials with unidirectional fiber orientation because of excess warping when heated on one side. Cross-plied materials did not exhibit this problem. In this configuration the specimen is subjected to the maximum radiative heat flux since it views the entire surface of the electrically heated hood. Measured radiative fluxes correlate well with that expected for a high emittance hemispherical source. (At a hood temperature of 1256^{o}K the measured radiative flux on the floor is nominally 150 kw/m^2.) A convective flux of approximately 40 kw/m^2 is obtained on the floor.

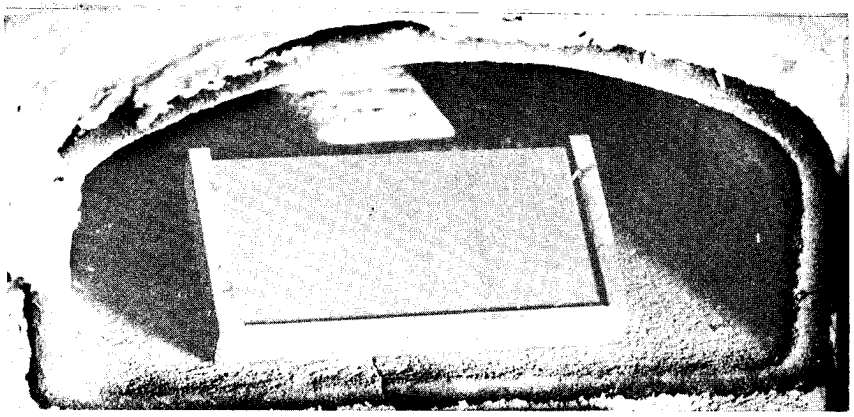
For the other orientations, specimen widths up to 124 mm and heights up to 80 mm can be accommodated. However, the recommended specimen size is 114.3x 63.5 mm. This permits the specimen to be mounted in a steel frame which restrains three of the specimen edges and also minimizes specimen warpage. The frame accommodates thickness up to 6.3 mm.



a. Vertically-mounted, perpendicular to flow



b. Vertically-mounted, parallel to flow



c. Mounted 45° to floor and flow with specimen frame

Figure 4. Specimen Configurations

The recommended orientation is with the specimen at 45 degrees to both the floor and the flow direction and with the unrestrained edge exposed to the combustion gas flow (Figure 4c). This configuration provides several advantages over the others evaluated:

a.) The specimen can be readily observed and photographed from the upstream furnace inlet.

b.) Calorimeters can be installed for direct calibration of radiative and convective flux. (This is not possible for vertically mounted specimens because the calorimeter sides and rear cannot be properly protected.)

c.) Fibers released from the heated surface are readily swept off the surface by the gas flow.

d.) Convective heat flux is maximized; levels of 57 kw/m^2 are readily achieved. (However, radiative fluxes are reduced to 2/3 that received in the floor mount location, because of a reduced view factor between the specimen and the radiative hood.)

Heat flux measurement is accomplished with Hi-Cal Corporation Model C-1300 and R-2040 Asymptotic (R) calorimeters and radiometers. The design of the two instruments differs only in that the radiometer has a transparent window over the sensing element which eliminates sensitivity to convective heating. The calorimeter element is sensitive to both the radiative and convective environments. The convective flux is deduced from the difference in the outputs of the two instruments. The manufacturer provides emissivity corrections for these instruments. Heat flux measurements are obtained by substituting the calorimeter block at the

specimen location. Calorimeter measurements are not possible during actual sample testing, but repeatability of test conditions is excellent and the substitution method is not considered a significant source of error.

The fuel source is commercial natural gas (methane) supplied through a Selas Corporation Model 20-CA combustion controller and a Model SH-4-FF burner nozzle (5 x 80 mm gas flow opening). Fuel and air are metered and pre-mixed by the controller. The burner nozzle mixture is injected through the floor of the furnace inlet duct where it is mixed with the air supplied by the forced draft system. The latter airflow rate is deduced from hot-wire anemometer measurement of the air velocity just downstream of the bell mouth in the inlet duct.

A range of gas flow and airflow rates were investigated during early testing in the facility and much of the early composite burn data were obtained with widely varying flow rates. Recent data have been obtained with two flow conditions which are recommended as standard conditions for future testing. The standard "lean" fire environment is achieved with 5 m^3/hr air and 180 scfh gas supplied from the Selas controller and mixed with 66 m^3/hr air flow from the inlet duct supply. The latter corresponds to a duct velocity of 1 m/s as measured by the anemometer and results in a lean air/fuel mixture ratio of 14 in the furnace test section. (Stoichiometric ratio is 9.8.)

The standard "rich" fire environment is achieved with the same fuel and air rates from the Selas, but with a reduced inlet duct velocity of 0.3 m/s. This provides 20 m^3/hr air flow in the inlet duct and an air/fuel ratio of 5. This mixture ratio results in a very sooty

fire and much soot deposited on the fiber trap wet filter along with the fibers. This soot is readily washed through the filter with a detergent. The variation in inlet duct air flow is achieved by varying the opening of bleed ports in the fiber trap chamber.

It should be noted that the duct velocities of 1 m/s and 0.3 m/s indicated are those of the room temperature air at the metering location. At the specimen location the velocities are estimated at 7.6 and 3.0 m/s, considering the blockage caused by the specimen and the expansion of the combustion gases upon heating to 1089°K (1800°F).

A flow agitation system is provided by injecting a small, pulsating flow (5 pulses per second) of argon parallel to the main combustion gas flow. This flow is directed at the center of the specimen heated surface with the intent of increasing the breakage and release of fibers which have lifted from the surface. However, the argon flow also imparts an overall pulsation to the combustion gas flow which is judged subjectively similar to the turbulence present in a large fire. The agitation flow velocity at the specimen location is estimated at 4.6 to 6.0 m/s based on an anemometer survey of the jet.

Use of the agitator greatly increases the fiber release rate for those materials which permit fibers to lift from the surface. Without agitation, the lifted fibers are consumed by combustion in a few seconds and relatively little material is collected downstream. With agitation, the fibers tend to fracture more readily and are carried out of the furnace before they are burned.

The orientation of the specimen at a 45 degree angle to the flow also enhances fiber release by the combustion gases sweeping the surface. For specimens oriented perpendicular to the flow, fractured fibers tend to remain trapped on the surface and are consumed by combustion.

III. REFERENCE COMPOSITE CHARACTERIZATION

A series of tests was conducted on a graphite-epoxy crossplied laminate composite, AS/3501-6, supplied by NASA. Two thicknesses were tested, a 24-ply $[\ (0, \pm 45, 90)_S\ ]_3$ 0.132 inch laminate identified as material B3 in the data tables; and a 32-ply $[0, \pm 45, 90_2 \mp 45, 0]_{2S}$ 0.176 inch laminate identified as B4.

Four specimen orientations were evaluated as follows:

a.) 124 x 124 mm panel, mounted flush to the floor of the test section.

b.) 124 x 61 mm panel, mounted vertically and parallel to the flow.

c.) 124 x 28 mm panel, mounted vertically and perpendicular to the flow.

d.) 124 x 61 mm panel, mounted 45° to both the floor and the flow.

The test condition parameters which were varied were:

a.) Radiation source temperature, $1089^{\circ}K$ and $1256^{\circ}K$.

b.) Air/fuel mixture ratio, 5 and 14 (rich and lean).

c.) With and without the pulsed argon jet agitation.

d.) Three edge restraint conditions; one edge free, three edges free, no free edges.

A total of 37 tests was conducted in this series. Results from selected tests are presented in Tables 2 through 4 to illustrate the significant test parameter effects. In general, these tests were successful in that substantial quantities of released fiber were collected and measured and a rational correlation was obtained between the fiber mass and the various test parameters.

As a result of this test series, it was concluded that the most significant conditions affecting fiber release are the edge restraint conditions and the degree of flow agitation. The air/fuel ratio and radiation source temperature seemed to be second order effects on the quantity of released fiber. In all cases, the quantity of fiber was relatively small; typically less than 1/10 of one percent of the original specimen weight.

The edge restraint condition was found to be very significant. Specimens were prepared with and without the edges potted with Sauereisen (a ceramic cement). With all edges unpotted, the specimens readily delaminated and had very little structural integrity. Imposing the argon agitation caused large pieces of laminate to become detached. Test results with this condition were understandably inconsistent. The specimen corners were particularly susceptible to laminate detachment.

The most consistent fiber release characteristics were obtained when the bottom and the vertical edges of the specimen were potted, and the specimen was oriented 45 degrees to the tunnel floor (as in Figure 4c). In this configuration, the argon agitation device was aimed at the center of the

TABLE 2

EFFECT OF AIR/FUEL RATIO

Test No.	Spec. No.	Agitation	Specimen Weight gm	Weight Loss Percent	Collected Fiber Weight gm	Collected Fiber Weight Percent *	Air/Fuel Ratio	Hood Temp °K	Restraint	Test Time Min.
39	B4-1	Yes	52.0	34	.021	.04	16	1256	Bottom & Sides	6
41	B4-1	Yes	52.0	33	.031	.06	9	1256	Bottom & Sides	6
31	B3-3	No	38.7	52	.032	.08	18	1256	Bottom & Sides	10
30	B3-3	No	39.0	42	Nil	---	8	1256	Bottom & Sides	10

All specimens at 45° to the floor and the flow direction.

*Percent of original specimen weight.

TABLE 3

EFFECT OF EDGE RESTRAINT AND TEST TIME

Test No.	Spec. No.	Agitation	Specimen Weight gm	Weight Loss Percent	Collected Fiber Weight gm	Collected Fiber Weight Percent*	Air/Fuel Ratio	Hood Temp ^{o}K	Edge Restraint	Test Time Min.
38	B3-2	Yes	38.0	45	0.123	0.32	16	1256	None	6
39	B4-1	Yes	52.0	34	0.021	0.04	16	1256	Bottom & Sides	6
40	B3-9	Yes	38.0	36	0.035	0.09	16	1256	All Edges	6
36	B3-3	Yes	38.0	57	0.069	0.18	16	1256	Bottom & Sides	10
34	B3-9	Yes	39.0	58	0.047	0.12	16	1256	All Edges	10

All specimens at 45^{o} to the floor and flow direction.

*Percent of original specimen weight.

TABLE 4

EFFECT OF AGITATION BY PULSED ARGON JET

Test No.	Spec. No.	Agitation	Specimen Weight gm	Weight Loss Percent	Collected Fiber Weight gm	Collected Fiber Weight Percent*	Air/Fuel Ratio	Hood Temp °K	Restraint	Test Time Min.
37	B3-9	Yes	38.2	65	.025	0.07	9	1256	Bottom & Sides	10
30	B3-3	No	39	42	Nil	---	8	1256	Bottom & Sides	10
33	B3-3	Yes (Severe)	37.8	60	.185	0.48	16	1256	Bottom & Sides	10
36	B3-3	Yes	38.0	57	.069	0.18	16	1256	Bottom & Sides	10
31	B3-3	No	38.7	52	.032	0.08	18	1256	Bottom & Sides	10

All specimens at 45° to the floor and flow direction.

*Percent of original specimen weight.

specimen and resulted in release of many small single fibers from this region. This is clearly observed in some of the photographs taken during the tests.

It was also apparent from visual observation and photography that the fiber material combusted readily. In several instances, a portion of a single laminate separated which was too large to be carried off by the gas flow. Typically, such fragments were nearly completely consumed within 15 seconds after separation. Obviously this phenomena is a source of uncontrolled variability in mass balance calculations. It was therefore concluded that a test configuration which prevented gross delamination of the specimens was necessary if consistent results were to be obtained.

As a result of this test series, a standard specimen configuration and environment was established for future testing (Table 5). A stainless steel frame(Figure 4c) was fabricated to permit restraining three specimen edges without resorting to the Sauereisen potting procedure. A standard specimen size of 114.3 by 63.5 mm (4½ by 2½ inches) was established, with a maximum thickness of 6.4 mm (¼ inch). Thinner specimens are held tightly by packing the excess space between frame and specimen with a small quantity of ceramic fiber insulation (Fiberfrax). The frame with specimen installed is mounted at 45 degrees to a ceramic plate which is elevated into position through a square opening in the floor of the furnace.

It should be noted that a change in the flow agitation method was made, effective for Test 59 and all subsequent tests. Early in the alternate materials testing discussed in the next section, difficulty was experienced with intermittent clogging of the agitator nozzle. This was invariably

TABLE 5

RECOMMENDED STANDARD TEST

Specimen Configuration

Size 114.3 x 63.5 mm ($4\frac{1}{2}$ x $2\frac{1}{2}$ inches), thickness variable.

Specimen mounted 45^{o} to floor, perpendicular to flow.

Three edges restrained.

Nominal Test Condition

Lean air/fuel mixture, 14:1.

Rich air/fuel mixture, $5\frac{1}{2}$:1.

Radiation source temperature, $1256^{o}K$ ($1800^{o}F$).

Radiation flux at specimen, 102 Kw/m^2 (9 Btu/ft^2-sec).

Convective flux at specimen, 57 Kw/m^2 (5 Btu/ft^2-sec).

Local velocity at specimen, 7.62 m/s (25 ft/sec) in lean environment, and 3.05 m/s (10 ft/sec) in rich environment.

Pulsed gas (argon) agitation of specimens.

followed by a pressure buildup in the argon supply tube and a blowout of the entire nozzle plug. This resulted in greatly increased agitation flow, since the orifice increased from the original 1 mm diameter to the full 4 mm diameter of the argon supply tube. In the interest of safety and test reliability, it was decided to eliminate the nozzle plug and reduce the argon supply pressure such that the same jet velocity was measured at the specimen location (55 m/s at a point 100 mm from the argon tube outlet). This resulted in an increased flow rate of argon and a somewhat more severe agitation condition than in earlier tests, as judged by visual observation and by test data for the reference composite before and after the change.

Table 6 presents a compilation of tests of the reference composite material which were performed after establishing the final recommended configuration and the revised agitation method. These data are considered the standard for comparison of the performance of the "alternate materials" of this program and for future screening of candidate materials in this facility.

An image analysis technique is available for identifying the length and diameter distribution of captured fibers. Tables 7 and 8 present the results for a random sampling of 150 fibers from a nominally stoichiometric burn of the reference composite material. The fiber sample was laminated between transparent plastic sheets. Each random fiber selection was made by placing the sample under a microscope and selecting the single fiber nearest the reticule cross-hair. Length and maximum diameter of this fiber were recorded. The process was repeated until a sampling of 150 fibers were completed. These data were then statistically analyzed by computer to obtain the results shown in the tables.

TABLE 6

FIRE TESTING OF LAMINATED GRAPHITE-EPOXY COMPOSITE IN FINAL RECOMMENDED TEST CONFIGURATION

Test No.	Specimen Panel No. (Note 2)	Specimen Weight gm	Fuel Mixture (Note 1)	Radiation Source Temp °K	Combustion Gas Temperature °K	Test Time Min.	Total Mass Loss Percent	Fiber Mass Collected Percent	Remarks
	8-ply, 3.4 mm thick composites								
61	B3-10	35.105	Lean	1256	1283	6	44	0.21	
60	B3-2	35.124	Lean	1256	1283	10	58	0.20	
65	B3-2	35.037	Rich	1267	1300	6	48	0.12	
	16-ply, 4.5 mm thick composites.								
62	B4-1	47.527	Lean	1256	1283	6	38	0.11	Surface ply vertical
63	B4-3	47.360	Lean	1256	1283	6	41	0.15	
64	B4-1	47.227	Lean	1256	1283	10	45	0.24	
66	B4-3	47.268	Rich	1256	1300	6	41	0.21	
94	B4-3	47.40	Rich	1256	1283	6	43	0.16	

Notes:
(1) "Lean" air/fuel ratio is 14:1. "Rich" is 5½:1.
(2) B3-2 and B3-10 are separate but identical panels, as are B4-1 and B4-3.

TABLE 7

LENGTH DATA FOR FIBERS RELEASED FROM REFERENCE COMPOSITE

Fiber Length mm	No. of Fibers Observed in Length Increment	Cumulative Total With Length Greater Than Value Indicated
		Percent
.315	5	100.0
.397	4	96.6
.500	7	93.7
.630	6	89.0
.794	7	85.2
1.00	6	80.6
1.26	19	76.8
1.59	14	64.3
2.00	15	54.8
2.52	14	44.3
3.17	14	35.6
4.00	15	26.1
5.04	8	16.2
6.35	13	10.8
8.00	3	1.9
10.00	0	0.0

Mean Length 2.57 mm

Median Length 2.25 mm

Standard Deviation 1.91 mm

TABLE 8

DIAMETER DATA FOR FIBERS RELEASED FROM REFERENCE COMPOSITE

Fiber Diameter Micron	No. of Fibers Observed in Diameter Increment	Cumulative Total With Diameter Greater Than Value Indicated
		Percent
.79	1	100.0
1.00	2	99.4
1.26	2	98.3
1.59	5	97.0
2.00	23	93.5
2.52	14	78.3
3.17	25	69.3
4.00	36	52.7
5.04	33	28.4
6.35	9	6.2
8.00	0	0.0

Mean Diameter 3.63 microns

Median Diameter 4.12 microns

Standard Deviation 1.35 microns

In the example shown, the mean fiber length was 2.57 mm with a standard deviation of 1.91 mm. The mean value of the maximum diameter was 3.63 microns, with a standard deviation of 1.35 microns. Since the original fiber diameter was nominally 8 microns, it was apparent that substantial diameter reduction by oxidation occurred. Many of the captured fibers taper near the ends, which is evidence of oxidation after fracturing of the fiber.

IV. ALTERNATE MATERIALS TESTING

To determine the applicability of the Model 25 facility as an effective screening test method, four different graphite fiber-reinforced composite materials were tested and a comparative evaluation of mass loss and fiber release characteristics was made. The materials were all supplied by NASA and are identified as follows:

Panel 78-157 - An 8-ply AS/3501 Woven Laminate

Panel 78-159 - A 16-ply AS/3501 Woven Laminate

Panel 53-1 - T300/Polyester Woven Laminate

Panel G3-5 - T300/178 (Hexcel Maleimide) Quasi-Isotropic Laminate

Panel E4-1 - AS/Xylok (Phenolic) Unidirectional Composite (Poor quality - many large cracks)

All the alternate material configurations were less subject to the problem of delamination and loss of laminate chunks from the surface than was the reference composite. However, with the exception of the AS/Xylok (phenolic) composite, these materials all released substantially greater masses of short fiber than did the reference composite. Test results are presented in Table 9.

TABLE 9

ALTERNATE MATERIALS TESTING

Test No.	Specimen Panel No.	Specimen Weight gm	Fuel Mixture (Note 1)	Radiation Source Temp °K	Combustion Gas Temp °K	Time Test Min.	Total Mass Loss %	Fiber Mass Collected (Note 2)	Remarks
	8-Ply AS/3501 Woven Laminate								
58	78-157	16.85	Lean	1256	1283	6	66	0.62	Excessive Agitation
59	78-157	16.94	Lean	1256	1283	6	55	0.27	
91	78-157	16.85	Rich	1256	1267	6	70	0.32	
54	78-157	16.23	Rich	1256	1267	6	44	0.13	Insufficient Agitation
	16-Ply AS/3501 Woven Laminate								
56	78-159	32.84	Lean	1256	1283	6	42	0.46	Excessive Agitation
57	78-159	32.63	Lean	1256	1283	6	37	0.40	
90	78-159	32.56	Rich	1256	1267	6	55	0.30	
	T300/Polyester Woven Laminate								
81	J3-1	55.05	Lean	1256	1289	6	58	0.52	Vigorous Combustion
88	J3-1	54.63	Rich	1256	1278	6	57	0.38	of Polyester Resin
	AS/Xylok (Phenolic) Unidirectional Composite								
82	E4-1	53.67	Lean	1256	1283	6	48	0.08	Very few fibers
92	E4-1	54.33	Rich	1256	1267	6	42	0.07	collected.
	T300/178 (Hexcel Maleimide) Quasi-Isotropic Laminate								
73	G3-5	44.34	Lean	1256	1306	6	50	0.59	
93	G3-5	44.03	Rich	1256	1283	6	44	0.33	

Note: (1) "Lean" air/fuel ratio is 14:1. "Rich" is 5½:1.
(2) Fiber mass given in percent of original specimen weight.

The AS/Xylok material (E4-1 panel) released far fewer fibers than any of the other materials. The post test structural character was also much better than the other materials. The test panel exhibited a characteristic tendency of unidirectional materials to warp badly during the first minute of heating. The restraint imposed by the specimen frame caused a number of partial fractures of the specimen along the fiber direction. However there was no loss of material as "chunks."

The T300/178 (Hexcel Maleimide material (G3-5 panel) was similar in structure and behavior to the reference composite. It delaminated readily in the fire and was subject to loss of chunks of laminate. It released a substantially greater quantity of small fibers than the reference composite. The structure of the residual material was poor.

The AS/3501 woven laminate composite delaminated readily and released far more small fibers than the reference material. The structural character of the residual material was delicate but not so much as the reference composite.

The T300/Polyester woven laminate composite (J3-1 panel) burned longer and more vigorously than the other materials. It did not delaminate and the residual material structure was good. However it released more small fibers than any of the other materials.

These tests satisfied the basic objective of demonstrating the capability of the facility and test method to identify differences in behavior and fiber release characteristics of a variety of different composite materials. It is concluded that the validity of the facility as a screening tool for evaluation of composites designed for improved fire performance is established.

V. FIBER OXIDATION TESTING

As noted in the reference composite testing, the loss of fiber mass by oxidation is an obviously significant phenomenon since burning of fiber material is readily observed visually. Comparisons of residual and original fiber masses in the reference composite characterization tests consistently indicated fiber masses of 20 percent or greater which were not accounted for. Although there was strong evidence of fiber combustion, there was concern for the possibility of free fiber loss from the facility. An experimental investigation was desired with the objective of quantifying the fiber combustion to determine if it was consistent with the observed discrepancy in the fiber mass balance. There was also a desire to obtain basic combustion rate data for possible correlation with data obtained from other facilities or from analytical predictions.

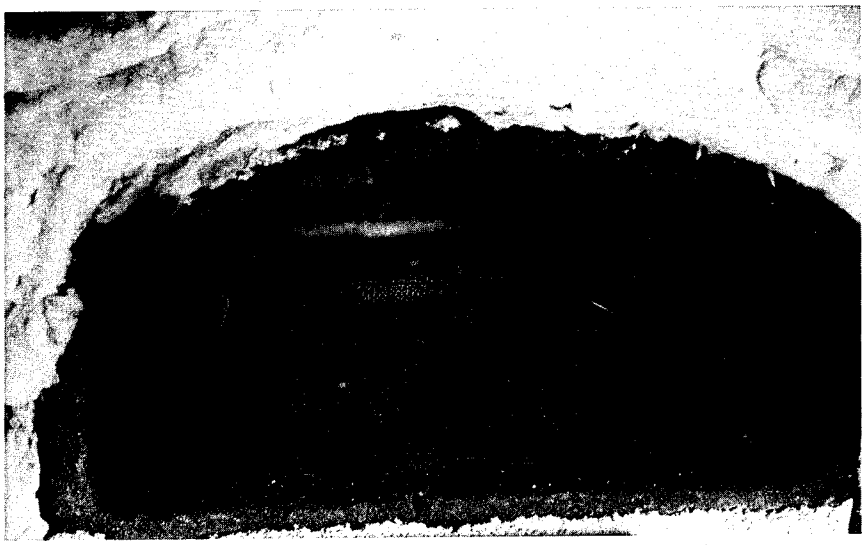
Several approaches for quantifying the oxidation phenomena were investigated. The basic technique was to expose fiber samples to a lean combustion environment (excess air) at temperatures consistent

with that in the simulated fire tests, but with a minimal agitation to prevent significant release of free fiber. Then any difference in pre-test and post-test fiber weight could be correctly attributed to combustion effects.

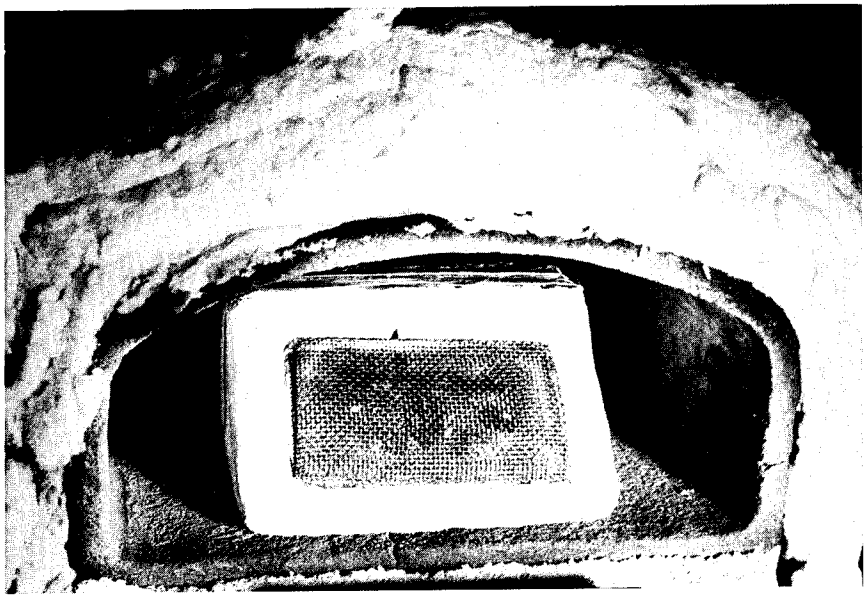
Several test configurations were attempted to achieve this condition. The initial approach placed fiber samples between Chromel alloy screens to permit exposure to the radiation and airflow environments without loss of fiber from the test zone. Another approach compared the mass losses from composite samples and identical samples which had been pre-cooked in an oven to remove the resin component ("biscuit sample"), with both samples evaluated in various fire test environments.

Initial tests were performed with the configuration of Figure 5a. Chopped A/S fibers, approximately 6 mm length, were placed between two Chromel alloy screens. This assembly was placed vertically in the furnace, perpendicular to the flow direction. The specimens were heated by radiation from the furnace hood and air was drawn through the screen assembly with the exhaust fan. The samples were exposed for varying lengths of time, using a separate fiber sample for each exposure and weighing the fiber mass before and after each test. No fuel was used in this series of tests. A test condition was established and specimens were exposed by inserting them into the floor opening, in sequence, without changing the test environment.

Tests 1-3 (Table 10) were with maximum airflow velocity (2 m/s) through the facility with a 1256^{o}K (1800^{o}F) radiation source temperature. The fibers combusted very rapidly in this environment after an initial time lag required to bring the screen assembly to a stabilized temperature.



a. Fibers mounted between Chromel screens



b. Fibers placed in firebrick cavity

Figure 5. Oxidation Test Configurations

Additional tests were performed with reduced flow velocity (Tests 4 and 5) and with reduced radiation source temperature of 1145°K (Tests 6 through 11). This environment slowed combustion to a rate at which fiber mass losses could be correlated with exposure time. However, large temperature gradients were apparent across the face of the screen. The screen temperature was increased in those areas where fiber clumps were in contact. Away from the fiber clumps, the screen was more effectively cooled by the air flowing through the assembly. This configuration is believed unreliable for combustion rate measurements because of the nonuniform sample temperature.

In tests 13 through 15, 35, and 37 the fibers were placed in a cavity in a holder made of firebrick (Figure 5b). The cavity was lined with Fiberfrax insulation and the fibers placed on the insulation. The cavity was then covered by a single Chromel screen to assure retention of the fibers. The screen did not contact the fibers. This was a better configuration than the other two. The screen heated rapidly to a stabilized and uniform temperature and fiber combustion rates were slow enough to be measurable by the sequential exposure technique described.

All the experiments of Table 10 were deficient for oxidation rate studies in that the sample temperatures are poorly defined and generally nonuniform across the specimen (this could be observed visually). However, the experiments showed conclusively that significant quantities of fiber material are consumed by combustion at temperatures and times which are consistent with those used in the composite fire tests.

Table 11 presents data from the cooked "biscuit" tests. In these experiments, panels of the reference composite material were cooked in an oven at 828°K to volatilize and drive off the epoxy resin component. The remaining fiber "biscuit" was then exposed to fire environments in the

TABLE 10
OXIDATION TESTING OF CHOPPED A/S FIBERS

Test No.	Specimen Weight gm	Percent Mass Loss	Test Time Min.	Radiation Source Temp. °K	Duct Velocity m/s	Duct Air Temp °K	A/F Ratio	Comment
		Flow thru Screen						
2	.3153	71	2	1256	2.0	922 (EST)	Infinite (1)	Difficult to obtain data because of rapid combustion at 1256°K.
3	.3260	~100	3	1256	2.0	922 (EST)	↓	
1	.3490	~100	4	1256	2.0	922 (EST)		
5	.3060	88	1.5	1256	1.5	1072		
4	.3314	100	1.5	1256	0.2	No Data	↓	
		Reduced Radiation - Flow thru Screen						
8	.3308	49	1.3	1144	1.5	839	Infinite	Relatively high velocity over specimen causes substantial cooling of specimen
6	.3145	55	2	1144	1.5	811	↓	
11	.3223	69	3.5	1144	1.5	811		
7	.3452	73	5	1144	1.5	894		
10	.3220	84	7	1144	1.5	922	↓	
		Screen Placed over Firebrick Cavity						
15	.3111	29	1	1144	1.5	906	Infinite	Best configuration – reduced velocity over specimen and radiation within cavity causes increased specimen temperature.
35	.3020	53	1.2	1144	1.5	978	↓	
37	.3001	75	1.5	1144	1.5	978		
14	.3162	82	1.5	1144	1.5	900		
13	.3024	90	2	1144	1.5	894	↓	

Note: These tests are in air only - no fuel gas burned.

TABLE 11
OXIDATION TESTING OF COOKED "BISCUITS"

Test No.	Specimen Weight gm	Percent Mass Loss	Test Time Min	Radiation Source Temp. °K	Duct Velocity m/s	Duct Air Temp °K	A/F Ratio
			Full "Biscuit" - No Fuel				
19	17.8731	9	2	1144	1.5	933	Infinite
17	13.9535	20	3	↓	↓	894	↓
16	16.4773	33	5	↓	↓	917	↓
18	17.2768	44	8	↓	↓	922	↓
			Single Ply From "Biscuit" - No Fuel				
24	.1438	31	0.75	1144	1.5	989	Infinite
23	.1630	44	1.00	↓	↓	989	↓
22	.1616	86	1.25	↓	↓	983	↓
21	.1593	96	1.75	↓	↓	989	↓
20	.2003	100	2.0	↓	↓	983	↓
			Single Ply - Rich Fire				
32	.1545	52	0.9	1144	0.3	1187	5.4
29	.1420	56	1.25	↓	↓	1194	↓
30	.1600	66	1.75	↓	↓	1192	↓
31	.1865	79	2.5	↓	↓	1200	↓

Model 25 facility to determine the quantity of fiber oxidized. It was intended to compare the total mass loss and fiber mass collected from a pre-cooked biscuit with that from a composite sample, both exposed to the same fire environment to determine if similar fiber oxidation losses were obtained from both configurations.

The initial biscuit tests (16 through 19) were performed on scrap ends of 8-ply B-3 reference composite panels. These were 125 mm by about 38 mm specimens and were angled at 45 degrees to the floor and airflow. No fuel was burned, but the facility exhaust system was left at the setting producing the lean condition of the conventional tests. This produced about a 50 percent greater air flow rate through the system (measured duct velocity 1.5 m/s compared to 1 m/s for the standard "lean" condition). No flow agitation was used.

The biscuit samples burned readily in this environment beginning at about 40 seconds of exposure. No specimen delamination or loss of fiber was apparent by visual observation, and negligible quantities were collected on the wet filter. Biscuit combustion losses ranging from 9 to 44 percent were obtained for exposure times of 2 to 8 minutes. In test 17, a 20 percent loss of fiber was obtained on a specimen exposed only three minutes (compared to a typical unaccountable fiber loss of about twenty percent in six minutes, obtained in many of the conventional tests in the standard lean environment).

The remaining tests of Table 11 were performed on single plies from pre-cooked biscuit laminates. These were placed in the firebrick cavity specimen holder described previously. A single ply was totally consumed in less than two minutes in the "no fuel" environment. Even in

the standard "rich fire" environment, the fiber biscuit material burned readily, with 79 percent of the mass of a single laminate consumed in 2½ minutes.

A comparison of the mass loss characteristics of a pre-cooked biscuit and a corresponding as-fabricated B-3 composite was made in both rich and lean standard fire environments, but without agitation (Table 12). For a 6-minute exposure, the residual specimen weights for both types of sample and both test environments were remarkably similar. In all four tests, the residual weight after fire exposure was approximately 40 percent less than the original composite weight. Assuming all the resin was lost (nominally 32 percent of the original composite weight), then the quantity of fiber loss attributable to combustion in these tests was approximately 8 percent of the original composite, or 12 percent of the original fiber weight. These figures are believed consistent with the 38 to 48 percent total mass losses obtained in the reference composite characterization tests of Table 8, with the somewhat higher losses of these latter tests being attributed to the effects of agitation. (With agitation, additional combustion losses occur because occasional "chunks" of laminate which break off and fall to the floor of the test section are completely consumed.)

In conclusion, it is believed that the "biscuit" experiments provide reasonable evidence that the substantial quantities of fiber which are not recovered in the fiber release experiments can correctly be attributed to fiber combustion.

TABLE 12

B-3 "BISCUIT" TESTS IN STANDARD FIRE ENVIRONMENTS

Test No.	Original Composite Weight gm	"Biscuit" Weight (Note 1) gm	Residual Weight After Fire Test gm	Test Time min.	Radiation Source Temp. °K	Air/Fuel Ratio	Total Mass Loss %	Fiber Mass Loss %	Remarks
43	34.99	27.95	22.01	6	1256	14	37	5	Biscuit Test
44	35.17	----	19.82	6	1256	14	44	12	Composite Test (Note 2)
48	35.01	27.8	20.96	6	1256	5½	40	8	Biscuit Test
49	35.17	----	20.89	6	1256	5½	41	9	Composite Test (Note 2)

Note: (1) "Biscuit" weight is measured after pre-cooking 10 minutes in oven at 828°K (1000°F).

(2) Fiber mass loss assumes an initial fiber material content of 68%.

VI. CONCLUSIONS

The recent modifications to the Model 25 fire simulation facility were demonstrated to be useful for evaluating the fiber dissemination characteristics of composite materials.

A comprehensive series of tests were performed on samples of an AS/3501-6 graphite-epoxy cross-plied laminate composite to evaluate the effects of various parameters on the quantity of fiber released. It was concluded that the most significant parameters were the degree of flow agitation and the configuration of the edge restraints on the specimen. These results indicated that a very significant mode of fiber release was the exposure of fiber ends at the specimen edges and subsequent fracture of these ends by the turbulence of the surrounding gas flow.

Consistent test results were obtained on specimens with only one edge exposed to the combustion gas flow. Results were much less repeatable with adjacent edges unprotected because of gross delamination and the fragile nature of the delaminated material. It was therefore found essential to provide a somewhat artificial structural integrity to the specimens by restraining at least three edges with either a metal frame or a ceramic potting material.

A specimen configuration was developed with three restrained edges which permitted evaluation of gas flow and heating parameters. Results of tests indicated that varying the air/fuel ratio from very rich to very lean had only a slight effect on the mass of fiber collected, but that significant loss of fiber by combustion occurred in both rich and lean environments. Experiments performed to specifically evaluate fiber combustion indicated that combustion losses of fiber material were typically of the order of twelve percent of original fiber weight, while released fiber quantities were of the order of one percent or less.

The effect of the severity of gas flow agitation was very significant. A standard condition was selected which was below a level which caused gross structural breakup of the residual fiber "biscuit" which remained after resin burnout.

The amount of fiber material lost by combustion and release was sensitive to the duration of exposure. A six-minute exposure was selected as standard. This was substantially longer than the time required for resin burnout (as much as two and one-half minutes for the thicker specimens) and sufficiently short that the thinner specimens did not burn completely through.

Results of the alternate materials testing indicated several trends. A subjective visual observation revealed that the woven composites released a significantly greater number of short single fibers than did the conventional quasi-isotropic laminate construction, although the mass of fiber collected was similar for both types of construction. A composite made with phenolic resin released fewer fibers and fiber mass than any of the epoxy resin composites.

The alternate materials testing phase of the program successfully demonstrated the capability of the facility and the test method to identify differences in behavior and fiber release characteristics of a variety of composite materials. It is concluded that the validity of the facility as a screening tool for evaluation of composites designed for improved fire performance is established.

The real fire performances of composites may be expected to differ quantitatively from the results obtained in the Model 25 facility because of the demonstrated significance of such parameters as gas flow agitation and material edge restraints. However, the general features of the standard test sequence are believed to adequately simulate the expected fire conditions for the purpose of material screening evaluations.

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15. Supplementary Notes

16. Abstract

A facility and test procedure were developed which are suitable for screening tests to evaluate fiber release characteristics of graphite fiber-reinforced composites in fire environments. Several types of composite construction were evaluated to establish the validity of the facility to identify variations in material performance. A specific composite currently in use in aircraft was extensively tested to examine the effects of variable test parameters on fiber release characteristics.

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